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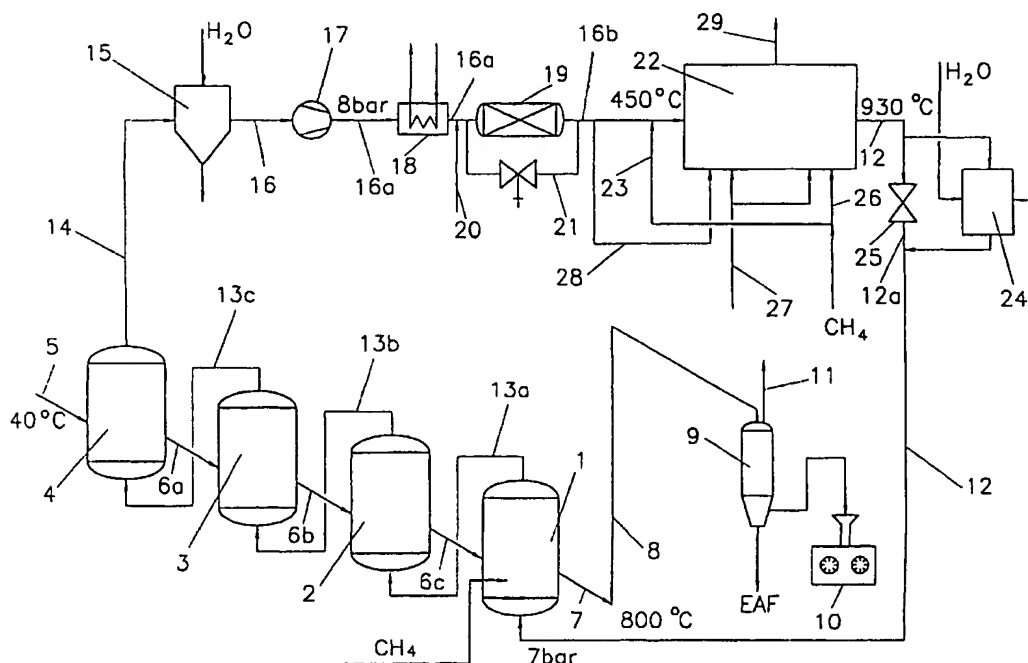
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(54) Titre : PROCÉDE DE REDUCTION DIRECTE DE MATERIAUX CONTENANT DE L'OXYDE DE FER

(54) Title: PROCESS FOR THE DIRECT REDUCTION OF IRON-OXIDE-CONTAINING MATERIAL



(57) Abrégé/Abstract:

The invention relates to a method for direct reduction of materials containing iron oxide using a reduction gas containing CO and H₂ in at least one fluidized bed reduction zone, whereby the used reduction gas containing CO₂ coming out of the at least one fluidized bed reduction zone is recirculated and a fresh reduction gas is produced by CO₂ reforming of the used reduction gas and a gas containing methane, especially natural gas. CO₂ reforming and direct reduction are carried out at a pressure of at least 4 bar overpressure in order to substantially prevent carbon formation and deposition and in order to keep the size of the reactor receiving the reduction zone small, whereby the reduction zone is exposed to an amount of reduction gas complying with metallurgical requirements.



Process for the direct reduction of
iron-oxide-containing material

5 The invention relates to a process for the direct
reduction of iron-oxide-containing material by means of
a CO- and H₂-containing reducing gas in at least one
fluidized-bed reduction zone, CO₂-containing, used
reducing gas which emerges from the at least one
fluidized-bed reduction zone being recirculated and
10 fresh reducing gas being produced by CO₂ reforming of
the used reducing gas and of a methane-containing gas,
in particular natural gas, and to an installation for
carrying out the process.

15 Processes in which CO- and H₂-containing reducing gas
is produced by what is known as steam reforming of
methane-containing gas and steam, the steam reforming
being carried out at high pressures and high
temperatures and hydrocarbons and steam being converted
20 into CO and H₂ by means of nickel catalysts in
accordance with the following reaction:

Steam reforming reaction: $\text{CH}_4 + \text{H}_2\text{O} \rightarrow \text{CO} + 3 \text{H}_2$

25 are known from the prior art, for example from
US-A-5,082,251.

In a CO shift reaction which follows the steam
reforming, the CO which is formed during the reforming
30 is then converted into CO₂ and H₂ in accordance with the
following equation:

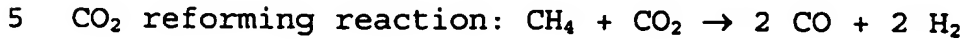
CO shift reaction: $\text{CO} + \text{H}_2\text{O} \rightarrow \text{CO}_2 + \text{H}_2$

35 The CO₂ usually then has to be removed from the
reformed gas, and the gas from which the CO₂ has been
removed has to be heated.

By contrast, in the case of CO₂ reforming, which is

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known, for example, from DE-A 196 37 180 and DE-A-195 17 766, not only steam is converted, but also CO₂, in accordance with the following equation:



The advantage of the CO₂ reforming is that there is no need for any removal of CO₂ or for any subsequent heating of the reducing gas to the desired reduction
10 temperature.

DE-A-196 37 180 has disclosed a process in which fine iron oxide particles are reduced by means of a CO- and H₂-containing reducing gas in a spouted bed and a
15 bubbling bed which is connected downstream of the spouted bed, the reducing gas being produced from the used CO-, CO₂- and H₂O-containing reducing gas by means of CO₂ reforming. The reforming and the direct reduction take place at low pressures of from 1.6 to
20 2.4 bar.

DE-A-195 17 766 has disclosed a process in which fine iron oxide particles are reduced in a plurality of circulating fluidized beds, which are connected in
25 series, likewise by means of a CO- and H₂-containing reducing gas, fresh reducing gas likewise, as in DE-A-196 37 180, being produced from the used CO-, CO₂- and H₂O-containing reducing gas by CO₂ reforming.

30 US-A-4,348,226 has disclosed a process in which off-gas from a reducing shaft furnace is mixed with natural gas, and the gas mixture is reformed in a heated reformer, and in which further natural gas is admixed with the reformed gas, and the gas mixture which is
35 then formed is subjected, in an unheated reactor, to an endothermic reforming reaction, fresh reducing gas being formed for the reduction shaft furnace. The sensible heat of the gas which has been reformed in the heated reformer is utilized in the second, endothermic

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reforming reaction, and the desired reducing-gas temperature is established.

5 It is known that CO₂ reforming takes place more successfully at lower pressures and that the reformer tubes can be designed to be thinner and therefore less expensive at low pressures.

10 The invention is based on the object of providing a process for the direct reduction of iron-oxide-containing material, in which CO- and H₂-containing reducing gas can be produced by CO₂ reforming of a methane-containing gas, in particular natural gas, and used reducing gas, in which, however,
15 the drawbacks of the known processes, which use a CO₂ reformer, such as the formation of carbon, deposits, large reactor diameters, etc., are to be avoided. The overall size of a reactor which accommodates the reduction zone is to be kept small, but at the same
20 time a quantity of reducing gas which satisfies the metallurgical requirements is to pass through the reduction zone.

According to the invention, this object is achieved by
25 the fact that the CO₂ reforming and the direct reduction are carried out at high pressure, preferably at a pressure of at least 4 bar superatmospheric pressure (5 bar absolute), in particular at a pressure of approximately 7 bar superatmospheric pressure. The
30 pressure range which is appropriate in a technical context in a process of this type is 6 to 8 bar superatmospheric pressure; the upper pressure limit is 15 bar superatmospheric pressure.

35 Surprisingly, it has been found that, in this way, many factors which have a disruptive effect on the reduction process, such as the formation of carbon and deposits, can be avoided in the fluidized-bed reduction zone.

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Furthermore, a sufficiently high supply of gas per unit volume of the reduction reactor to satisfy the metallurgical requirements is provided for the reduction, so that the reactors which accommodate the fluidized-bed reduction zones can be of smaller dimensions. Nevertheless, a sufficient gas throughput is still ensured. Moreover, the reduction potential of the reducing gas is higher.

Furthermore, iron sponge which is produced during the direct reduction of iron-oxide-containing material can advantageously be fed by pneumatic conveying by means of the reducing gas to be briquetted, so that a briquetting device which is used for the briquetting can be arranged next to a direct reduction device which is used for the direct reduction, with the result that the overall size of the entire installation for carrying out the process according to the invention can be kept small.

The advantage of the process according to the invention is that the CO_2 which is present in the used reducing gas does not have to be removed, but rather is used directly for the production of fresh reducing gas. Compared to known direct reduction processes, for example that described in US-A-5,082,251, which was mentioned in the introduction, in which the reducing gas is produced by steam reforming, without the steam reformer being connected into the reducing-gas circuit, connecting the CO_2 reformer into the reducing-gas circuit means that a lower specific flow of reducing gas is required for the direct reduction; the specific flow of reducing gas is understood to mean the flow rate of freshly supplied reducing gas based on the material which is to be reduced.

It is preferable for the used reducing gas to be subjected to a CO shift reaction at least in part prior to the reforming. In this way, the CO is converted into

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CO₂ and H₂ by means of steam in accordance with the following equation:

CO shift reaction: $\text{CO} + \text{H}_2\text{O} \rightarrow \text{CO}_2 + \text{H}_2$.

5

The CO content of the gas supplied to the reformer is advantageously minimized in the process, and the CO/CO₂ ratio is set.

- 10 On account of a high CO content in the reducing gas, in particular if the gas which is to be reformed already contains CO, problems caused by metal dusting, which is understood as meaning destruction of the metallic parts of the installation by CO, may occur in metallic parts
15 of the installation. If the gas which is to be reformed, should it contain CO, is subjected to a CO shift reaction, metal dusting can be substantially avoided.
- 20 If the H₂O content of the CO₂- and CO-containing gas is not high enough for a CO shift reaction, steam is advantageously added to the CO shift reaction.

On account of the once-through operation, which is
25 understood as meaning the fact that the reformer is connected directly into the reducing-gas circuit, without any devices which have a significant influence on the temperature and composition of the reducing gas being provided between the reformer and a reduction
30 reactor which accommodates the fluidized-bed reduction zone, there are fewer possible ways of adjusting the reducing-gas quality than if the reformer is connected outside the reducing-gas circuit. According to WO-A-96 00304, which, like US-A-5,082,251, has
35 disclosed a direct reduction process using a steam reformer connected outside the reducing-gas circuit, there are, for example, possible ways of setting the reducing-gas quality by changing the way in which the reformer operates, by changing the extent to which CO₂

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is scrubbed out of the reformed gas and/or used reducing gas, etc.

5 With the aid of the CO shift reaction which is provided according to a preferred variant of the process according to the invention, it is possible even when using once-through operation for the gas ratios required for the reforming and the direct reduction to be set as required, i.e. for the CO/H₂ ratio to be
10 varied or the CO content to be reduced according to the specific requirements.

According to a further preferred embodiment, the used reducing gas is compressed prior to the reforming,
15 preferably to a pressure of approximately 8 bar superatmospheric pressure.

It is preferable for the waste heat of the reforming to be used to preheat air, H₂O, natural gas, etc.

20 The used reducing gas is advantageously compressed prior to the CO shift reaction, preferably to a pressure of approximately 8 bar superatmospheric pressure.

25 The used reducing gas is expediently heated prior to the reforming and prior to the optional CO shift reaction.

30 The present invention also relates to an installation for carrying out the process according to the invention, having at least one fluidized-bed reactor, which accommodates a fluidized-bed reduction zone, a feed line for feeding a CO- and H₂-containing reducing
35 gas to the fluidized-bed reactor and a gas discharge line for discharging used reducing gas, which leads from the fluidized-bed reactor to a CO₂ reformer in order to produce the CO- and H₂-containing reducing gas from a methane-containing gas, in particular natural

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gas, and the used reducing gas, the CO₂ reformer being line-connected to the fluidized-bed reactor via the feed line.

5 According to the invention, this installation is characterized in that there is a compression device for compressing the gas which is supplied to the fluidized-bed reactor to a high pressure, preferably to a pressure of at least 5 bar superatmospheric pressure,
10 in particular to a pressure of approximately 8 bar superatmospheric pressure, upstream of the CO₂ reformer.

It is preferable for a CO shift reactor to be provided
15 upstream of the CO₂ reformer for used reducing gas. The feed line for steam may in this case open out upstream of the CO shift reactor into a feed line for the CO₂-and, if appropriate, CO-containing gas and/or into the CO shift reactor itself.

20

According to an even more preferred embodiment, the compression device for compressing the used reducing gas is provided upstream of the CO shift reactor.

25 In the installation according to the invention, it is preferable for at least three, and in particular preferably four, fluidized-bed reactors which are connected in series to be provided.

30 To accurately set the chemical composition of the reducing gas for optimum efficiency of the CO₂ reformer, the CO shift reactor can expediently be bypassed by means of a bypass line for the used reducing gas.

35

It is advantageous for a line which supplies a CH₄-containing gas, in particular natural gas, to open out into the gas line which supplies used reducing gas to the CO₂ reformer.

The installation according to the invention is expediently characterized by a heating device for the cleaned and compressed used reducing gas.

5

The invention is explained in more detail below with reference to the drawing, in which Figures 1 and 2 in each case illustrate a preferred embodiment of the invention, identical components in each case being
10 provided with identical reference symbols.

Figure 1 shows four fluidized-bed reactors 1 to 4 which are connected in series and each accommodate a steady-state fluidized bed, iron-oxide-containing
15 material, such as fine ore, being supplied via an ore feed line 5 to the uppermost fluidized-bed reactor 4, in which heating to reduction temperature and, if appropriate, preliminary reduction take place, and then being passed from fluidized-bed reactor 4 to
20 fluidized-bed reactors 3, 2 and 1 via delivery lines 6a to 6c. The fully reduced material (iron sponge) is fed, via a discharge line 7 and a riser 8, which is understood as meaning a substantially vertical section of pipe which has a refractory lining and is used to
25 convey the iron sponge pneumatically upwards by means of the reducing gas, to a storage hopper 9 and, from there, to a briquetting device 10, in which the iron sponge is hot-briquetted. If appropriate, the reduced material is protected from reoxidation during the
30 briquetting by an inert-gas system (not shown) or is fed to an electric arc furnace situated below.

The reducing gas which is used to convey the iron sponge through the riser 8 is extracted and expanded
35 via a line 11 and is then fed for further use, for example for heating purposes (not illustrated). The use of a riser 8 has the advantage that the briquetting device 10 can be arranged next to the reduction device formed from the fluidized-bed reactors 1 to 4, with the

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result that the overall height of the entire installation can be lowered. A further possibility (not illustrated) of conveying the iron sponge into the storage hopper 9 without using a riser 8 consists in the lowermost fluidized-bed reactor 1 being arranged at a height which is such that the iron sponge can flow into the storage hopper 9, which is arranged at a lower level, by means of the force of gravity; in this case, however, the drawback of a greater overall height of the entire installation has to be accepted.

Before the iron-oxide-containing material is introduced into the first fluidized-bed reactor 4, as seen in the direction of flow of the material, it is subjected to a preparation treatment, such as a drying treatment (not illustrated in more detail).

Reducing gas is fed to the lowermost fluidized-bed reactor 1 via a feed line 12, is carried from fluidized-bed reactor 1 to fluidized-bed reactors 2, 3 and 4 via lines 13a to 13c in countercurrent to the flow of the material which is to be reduced and is extracted via a gas discharge line 14 as used reducing gas. By way of example, the reducing gas flows into the lowermost fluidized-bed reactor 1 at a temperature of approximately 800°C and a pressure of approximately 8 bar absolute and leaves the uppermost fluidized-bed reactor 4 as used reducing gas at a temperature of approximately 550°C and a pressure of approximately 6 bar absolute.

The used reducing gas is cooled and scrubbed in a cooler/cleaner 15, where dust and steam are removed. The cooled and cleaned gas, which according to the embodiments illustrated is passed through a circuit, is then fed to a compressor 17 via a line 16. In the compressor 17, the used reducing gas is compressed, for example to a pressure of approximately 8 bar. Following the compressor 17 there is a heating device 18, which

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is used to heat the used reducing gas, which has been greatly cooled during the cleaning by the cooler/cleaner 15, back up to a temperature which it needs for a CO shift reaction. The used reducing gas which has been heated in this way is then fed via the line 16a to a CO shift reactor 19, in which the CO which is present in the used reducing gas is partly converted, by means of steam, to CO_2 and H_2 . In the exemplary embodiment illustrated in Fig. 1, steam is fed via a feed line 20 into the line 16a by means of which the used reducing gas is carried to the CO shift reactor 19. However, the steam may also, by way of example, be fed directly into the CO shift reactor 19. In the CO shift reactor 19, the CO which is present in the used reducing gas is (partially) converted into CO_2 and H_2 by means of steam.

The provision of the CO shift reactor 19 on the one hand advantageously increases the CO_2 content of the gas which is fed to the CO_2 reformer, which promotes the reformer reaction, and, on the other hand, reduces the CO content, with the result that metal dusting, i.e. the destruction of metallic parts of the installation by CO, is substantially avoided. In addition, the CO shift reactor 19 results in more possible ways of setting the desired reducing-gas quality. The gas ratios required for the reforming and the direct reduction can be set according to the particular requirements, i.e. the CO/H_2 ratio can be varied and/or the CO content can be reduced according to requirements.

The CO shift reactor 19 can be bypassed by means of a bypass line 21, resulting in a wide range of possibilities for setting the desired reducing-gas quality, for example as a result of a partial quantity of the used reducing gas being fed directly to the CO_2 reformer 22 without being passed through the CO shift reactor 19.

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In the CO₂ reformer 22, the gas which is supplied via the line 16b, if appropriate prior to heating, is reacted together with methane-containing gas, in the example illustrated natural gas, which is supplied via a line 23, so that CO and H₂ are formed.

The reformed gas leaves the CO₂ reformer for example at a temperature of approximately 930°C. To allow it to be used as fresh reducing gas, the reformed gas still has to be heated to the desired reducing-gas temperature. In the exemplary embodiment illustrated, the reformed gas which is extracted from the CO₂ reformer 22 via a line 12 is in part guided via a cooler 24 and the remaining part is guided via a line 12a which bypasses the cooler and has a valve 25, during which process a reducing-gas temperature of approximately 800°C is established.

The CO₂ reformer 22 is heated by burning natural gas, which is supplied via a line 26, with an oxygen-containing gas, such as air, which gas is supplied via a line 27. Part of the used, heated reducing gas can be branched off via a line 28 and can likewise be burned with an oxygen-containing gas, such as air, in order to heat the CO₂ reformer 22. The combustion off-gases which are formed in the process are extracted from the CO₂ reformer 22 via a line 29.

The high pressure in the reducing-gas circuit, for example approximately 7 to 8 bar absolute upstream of the CO₂ reformer 22 and approximately 6 to 7 bar before the gas is introduced into the lowermost fluidized-bed reactor 1, allows all the internal fittings (lines, fluidized-bed reactors) to be of correspondingly small dimensions. Furthermore, the formation of carbon and deposits is substantially avoided in all components. Finally, a riser 8 may advantageously be used to convey

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the reduced material to the briquetting device 10, as has already been explained in more detail above.

According to the embodiment illustrated in Fig. 2, the
5 used reducing gas, after it has been heated in the heating device 18, is fed directly to the CO₂ reformer 22, with the result that the installation is simplified, but there is not such a wide range of possibilities for influencing the composition of the
10 reducing gas leaving the CO₂ reformer as there are in the embodiment illustrated in Fig. 1.

Chemical compositions of the gases, temperatures and pressures in accordance with the exemplary embodiment
15 illustrated in Fig. 1 are explained in more detail in the example which follows (pressure details are in bar absolute).

A) Flow of ore

20 Ore introduced into the fluidized-bed reactor 4 via the ore feed line 5:

Temperature: approx. 50°C, ore weight based on the product approx. 1.44.

25 Composition: hematite (Fe₂O₃) with a pure iron content of approx. 67%, grain size up to at most 12.5 mm.

Ore discharged from the fluidized-bed reactor 1 via the discharge line 7:

30 Temperature: approx. 800°C, reduced ore

Composition: total iron content approx. 93% (Fe), metallization 92%

C = 1.5 - 2.5%

Grain size: up to at most 6.3 mm

35 The reduced ore is conveyed for briquetting 10 via the riser 8.

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B) Gas flow

Gas introduced into the fluidized-bed reactor 1 via the line 13:

Pressure: approx. 7 bar superatmospheric pressure

5 Temperature: approx. 800°C

Reducing-gas composition: CO: 21.7%

CO₂: 3.2%

H₂: 57.2%

H₂O: 5.6%

10 CH₄: 6.2%

N₂: 6.1%

Gas discharge of the used reducing gas from the fluidized-bed reactor 4 via the gas discharge line 14:

15 Pressure: approx. 5 bar superatmospheric pressure

Temperature: approx. 550°C

Gas composition: CO: 15.4%

CO₂: 8.8%

20 H₂: 46.5%

CH₄: 4.4%

H₂O: 18.3%

N₂: 6.5%

25 Dust content in the gas: approx. 27 kg/t of product, with 9.5 g/m³n.

Deposition of the dust through reducing-gas scrubber 15 (also referred to as cooler/cleaner):

30

Used reducing gas after scrubber 15:

Pressure: approx. 4 bar superatmospheric pressure

Temperature: approx. 40°C

35 Dust content: 27.3 g/t of product with approx. 10 mg/m³n.

Used reducing gas after the compressor 16:

Pressure increase to approx. 8 bar superatmospheric pressure

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Temperature: approx. 100°C

Used reducing gas after the heating device 18:

Pressure: approx. 7.8 bar superatmospheric pressure

5 Temperature: approx. 350°C

Input into the CO shift reactor 19:

Pressure: approx. 7.8 bar superatmospheric pressure

Temperature: approx. 350°C

10

Gas composition:	CO:	14.0%
	CO ₂ :	8.0%
	H ₂ :	42.4%
	H ₂ O:	26.6%
15	CH ₄ :	4.0%
	N ₂ :	5.2%

Used reducing gas after the CO shift reactor 19:

Pressure: approx. 7.5 bar superatmospheric pressure

20 Temperature: approx. 450°C

Entry of the used reducing gas into the CO₂ reformer 22
(after CH₄ has been admixed):

Pressure: approx. 7.5 bar superatmospheric pressure

25 Temperature: approx. 450°C

Gas composition:	CO:	4.4%
	CO ₂ :	13.6%
	H ₂ :	43.9%
	H ₂ O:	14.9%
30	CH ₄ :	17.5%
	N ₂ :	5.8%

Reducing-gas discharge from CO₂ reformer 22 via the
line 12:

35 Pressure: approx. 7 bar superatmospheric pressure

Temperature: approx. 930°C

Gas composition:	CO:	22.6%
	CO ₂ :	3.3%
	H ₂ :	59.5%

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H ₂ O:	6%
CH ₄ :	2.4%
N ₂ :	6.1%

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Patent Claims

1. Process for the direct reduction of iron-oxide-containing material by means of a CO- and H₂-containing reducing gas in at least one fluidized-bed reduction zone, CO₂-containing, used reducing gas which emerges from the at least one fluidized-bed reduction zone being recirculated and fresh reducing gas being produced by CO₂ reforming of the used reducing gas and of a methane-containing gas, in particular natural gas, characterized in that the CO₂ reforming and the direct reduction are carried out at high pressure, preferably at a pressure of at least 4 bar superatmospheric pressure (5 bar absolute), in particular at a pressure of approximately 7 bar superatmospheric pressure.

2. Process according to Claim 1, characterized in that the used reducing gas is subjected to a CO shift reaction at least in part prior to the reforming.

3. Process according to Claim 2, characterized in that steam is added to the used reducing gas before and/or during the CO shift reaction.

4. Process according to one of Claims 1 to 3, characterized in that the used reducing gas is compressed prior to the reforming, preferably to a pressure of approximately 8 bar superatmospheric pressure.

5. Process according to one or more of Claims 2 to 4, characterized in that the used reducing gas is compressed prior to the CO shift reaction, preferably to a pressure of approximately 8 bar superatmospheric pressure.

6. Process according to one or more of Claims 1 to 5, characterized in that the used reducing gas is heated

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prior to the reforming and prior to the optional CO shift reaction.

7. Installation for carrying out the process according to one of Claims 1 to 6, having at least one fluidized-bed reactor (1 to 4), which accommodates a fluidized-bed reduction zone, a feed line (12, 13) for feeding a CO- and H₂-containing reducing gas to the fluidized-bed reactor (1 to 4) and a gas discharge line (14, 16, 16a, 16b) for discharging used reducing gas, which leads from the fluidized-bed reactor (1 to 4) to a CO₂ reformer (22) in order to produce the CO- and H₂-containing reducing gas from a methane-containing gas, in particular natural gas, and the used reducing gas, the CO₂ reformer (22) being line-connected to the fluidized-bed reactor (1 to 4) via the feed line (12, 13), characterized in that there is a compression device (17) for compressing the gas which is supplied to the fluidized-bed reactor (1 to 4) to a high pressure, preferably to a pressure of at least 5 bar superatmospheric pressure, in particular to a pressure of approximately 8 bar superatmospheric pressure, upstream of the CO₂ reformer (22).

8. Installation according to Claim 7, characterized in that a CO shift reactor (19) is provided upstream of the CO₂ reformer (22) for used reducing gas.

9. Installation according to Claim 8, characterized in that a feed line (20) for steam opens out into the CO shift reactor (19) or into the gas line (16a) which carries used reducing gas and opens out into the CO shift reactor (19).

10. Installation according to one of Claims 7 to 9, characterized in that the compression device (17) for compressing the used reducing gas is provided upstream of the CO shift reactor (19).

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11. Installation according to one or more of Claims 7 to 10, characterized in that at least three, preferably four, fluidized-bed reactors (1 to 4) which are connected in series are provided.

5

12. Installation according to one or more of Claims 8 to 11, characterized in that the CO shift reactor (19) can be bypassed by means of a bypass line (21) for the used reducing gas.

10

13. Installation according to one or more of Claims 7 to 12, characterized in that a line (23) which supplies a CH₄-containing gas, in particular natural gas, opens out into the gas line (16b) which supplies used
15 reducing gas to the CO₂ reformer (22).

14. Installation according to one or more of Claims 7 to 13, characterized by a heating device (19) for the cleaned and compressed used reducing gas.

